## ASSOCIATION OF MINERAL MATTER WITH THE ORGANIC COAL MATRIX

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### ABSTRACT

Advances in the quantitative assessment of the association of mineral particles with the organic coal matrix have been made recently at the Ames Laboratory. In addition to routine analysis of mineral matter for particle size and mineral phase, coal particles are classified according to the mass fraction of the various minerals found in cross sections of the particles. Particles are also classified according to the relative amount of mineral matter and coal present on their surface. Examples are given of the resulting distributions for individual minerals showing their liberation, and results are related to coal recovery and ash reduction of the sample during cleaning.

### INTRODUCTION

Variations in the characteristics of mineral matter in coal have a significant bearing on the effectiveness of removal of mineral matter during cleaning. The size distributions of the mineral particles play a significant role in determining cleanability since it is generally easier to remove the larger mineral particles. However, it is the association of mineral particles with the organic coal matrix that ultimately determines the cleaning potential. On occasion, certain minerals can be preferentially liberated and then physically removed while others remain associated with the coal matrix.

In practice, coal is not ground to a size at which all mineral matter is liberated from coal; it would not be economically feasible. Rather, coal is processed only to the extent that is necessary to comply with applicable environmental regulations. In this context, it is more important to know to what extent the coal and minerals are still associated.

In the last few years, image analysis techniques have been adapted to the in-situ characterization of the association of individual minerals with coal. Scanning electron microscopy (SEM) is used to observe coal and mineral particles in cross section, energy-dispersive x-ray analysis (EDX) is used to determine the elemental composition of the mineral particles, and automated image analysis (AIA) is employed to characterize a sufficient number of particles for reproducibility. Such techniques have been in use in the mineral industry for many years (1,2). However, the application of these techniques to coal has lagged, partly due to the inability to resolve coal particles from the mounting media. Conventional epoxy resins do not exhibit contrast with coal particles. Therefore, the use of carnauba wax was developed as an alternative and effective medium (3).

## METHODOLOGY

Two sets of coal samples were chosen to illustrate applications of AIA to coal processing. The first coal was a 200-mesh sample of Williams Fork Q bed coal from Moffat County, Colorado. The coal is ranked as sub-

bituminous A, with 15.3% moisture, 4.18% ash, and 0.45% total sulfur. The coal was subjected to bench-scale float-sink cleaning at 1.6 sp.gr. in a centrifuge. Samples of the raw coal and the float and sink fractions were then collected for analysis. The full procedure has been described extensively elsewhere (4). The second sample was a 325-mesh sample of Upper Freeport coal with 1.3% moisture, 9.88% ash, and 1.56% total sulfur. The coal was cleaned in two separate tests by float-sink and by froth flotation, as described eleswhere (5). Only samples of the raw coal were available for AIA.

Coal samples with their included mineral matter were prepared for image analysis by mixing samples of the dry coal with polyethylene powder (as a diluent) and molten carnauba wax. Because SEM-AIA is often used to explain behavior under a specific set of processing conditions, samples are typically prepared in the same size in which they are received. Pellets were then cut to expose a vertical cross section and polished using standard petrographic procedures. They were then coated with 150 Å of carbon to provide a conductive surface.

Samples were examined with an electron beam of 15 keV and 0.7 nA at magnifications of 200-500 using the backscattered electron (BSE) signal. Use of the BSE signal permits relatively easy differentiation of minerals and coal from each other and from the carnauba wax using simple brightness thresholds. Coal and mineral particles were characterized for area and perimeter, and information on the relationship of adjoining particles with each other was preserved in the stored data. The amount of surface for each particle in contact with coal, mineral matter, and/or mounting media was recorded. X-ray spectra were then collected for 4 seconds for each of the mineral particles. The integrated intensities for 20 elements were compared with a previously prepared table listing ranges of elemental intensities characteristic of minerals found in coal in order to identify the particles (6,7). Handbook values of mineral densities were then used

of more direct interest in coal preparation.

Results involve very detailed information for the composite coal/mineral particles and their component parts (i.e., size, identification, and surface associations). The analyst can then prepare tables showing the distributions of the sample mass as a function of the appropriate characteristic. These distributions can then be related to processing behavior. Examples of such distributions are given below.

to convert the results from area fractions to weight fractions, which are

# RESULTS AND DISCUSSION

A typical distribution of mineral matter according to particle size and mineral phase is given in Figure 1 for the major phases in the raw sample of Williams Fork coal. Such particle size distributions relative to the size of the coal particles can be used to predict the ash reduction potential, since larger mineral particles are generally more easily removed, while small mineral particles are likely to be associated with the organic matrix and to appear with the clean coal product. As seen in Figure 1, most of the mineral matter in this coal is quite fine and is thus expected to be rather difficult to remove. However, it is not unusual to find minerals, such as those that occur as cleat fillings, that are readily liberated and then removed by cleaning. Such is also the case with the pyrite in this coal. Pyrite particles show a bimodal size distribution in Figure 1. The larger particles are likely candidates for

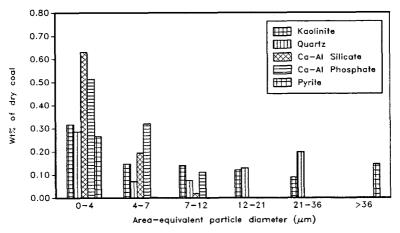


Figure 1. Distribution of selected minerals in raw Williams Fork coal as a function of area-equivalent particle diameter (μm).

removal; however, analysis of the 1.6 sp.gr. float fraction of the coal shows that even small grains of pyrite were removed, implying that they were very well liberated. Such cases point out the need to perform some form of association analysis in order to better characterize the coal.

As one alternative, we have chosen to express the coal-mineral association results in terms of the weight fraction of mineral matter in the particle, as determined from the cross section. This corresponds with the so-called grade distributions used in the mineral industry (1,2). Such a distribution for the same sample of Williams Fork coal is shown in Table 1. The results can also be plotted as done in Figure 2 to show the amount of sample of the indicated grade. Samples with good liberation of minerals from coal show a wide separation between coal-rich material on the left and mineral-rich material on the right side of the figure. In this sample there is much mineral matter found across the entire range of grades, indicating that liberation of mineral matter is not complete at this particle size, and that physical cleaning would therefore be difficult.

The cumulative amount of coal and associated mineral matter, observed in Figure 2, can be used to estimate coal recovery and its anticipated ash content during a density-based separation. However, such a correlation is complicated since the AIA-observed mineral content does not directly reflect particle density. Still, such distributions often show dramatic differences between various coal samples. Similar figures for float or sink fractions can reveal the misplacement of coal- or mineral-rich particles to the wrong fraction so that improvements can be made in the process.

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Table 1. Distribution of coal and mineral phases in raw Williams Fork coal as a function of particle mineral matter content.

Coal %	0-9	10-19	20-29	30-39	40-49	50-59	60-69	70-79	80-89	90-99	100	Total
Coal Pyrite Kaol. Quartz Mont. Other	0.39 0.07 0.23 0.40	0.02 0.09 0.06 0.01	0.00 0.11 0.20 0.04	0.00 0.13 0.03 0.02	0.00 0.06 0.08 0.01	0.00 0.10 0.02 0.03	0.00 0.05 0.03 0.02	0.00 0.08 0.03 0.02	0.00 0.06 0.04 0.02	0.00 0.06 0.04 0.03	54.36 0.00 0.00 0.00 0.00	0.41 0.81 0.76 0.61
	2.97											100.00

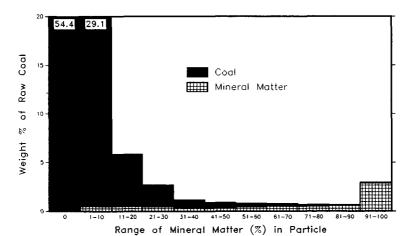


Figure 2. Distribution of coal and mineral matter in raw Williams Fork coal as a function of the particle mineral matter content.

The data of Table 1 can also be plotted for the individual minerals to show variations in the modes of their association, as shown in Figure 3. This figure dramatically shows the difference between pyrite and the other minerals in their association with coal. Pyrite is found exclusively in particles containing more than 80% mineral matter, while the other minerals are associated with particles containing a wide range of mineral matter. From this figure, it appears that pyrite should be easily removed during float-sink separation, which proved to be the case in actual separations, both on a laboratory scale (5) and in practice. Representatives from the mine which produces this coal confirmed that the pyrite occurs as cleat fillings and is rather easily removed (8). Similar

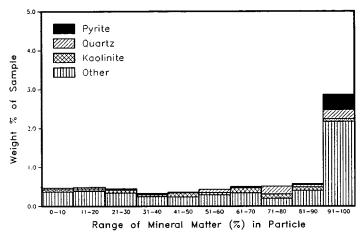


Figure 3. Distribution of minerals in raw Williams Fork coal as a function of particle mineral matter content.

conclusions might be reached by routine manual observation of the polished sections or from operating experience; however, these AIA-SEM techniques permit the measurements to be quantified for comparisons among coals.

In addition to expressing coal-mineral association as a function of particle mineral matter content, as described above using the Williams Fork coal as an example, our recent efforts have emphasized determining association based on particle surfaces. While the previous distributions provide an indication of the probable cleaning behavior of a coal in a density-based process, they do not lend much insight into cleaning behavior during surface-based processes such as froth flotation or oil agglomeration. For such processes it would be better to have results expressed in terms of the proportion of coal (or minerals) present on the surface of the particles. If it were possible to relate floatability to the amount of coal on the surface, then it may be possible to relate cleanability to the AIA-SEM results.

Figures 4 and 5 show the coal-mineral association for Upper Freeport coal based on the mineral weight fraction and the mineral surface fraction of the particles. There is considerable difference between the two figures. While Figure 4 shows that about 74% of the mineral matter is present in particles containing more than 50% mineral matter (i.e., less than 50% coal), Figure 5 indicates that only 10% of the mineral matter is found in particles with less than 50% of the surface covered by coal. Indeed, about 75% of the mineral matter is found in particles with more than 80% coal on the surface. These results indicate that density-based processes (e.g., float-sink) should be able to remove significant amounts of mineral matter, while surface-based processes will likely be unable to

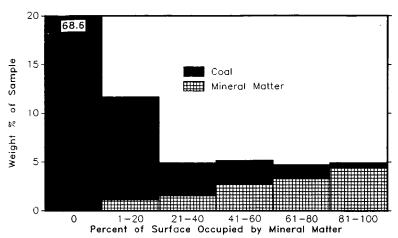


Figure 4. Distribution of coal and mineral matter in raw Upper Freeport coal as a function of particle mineral matter content.

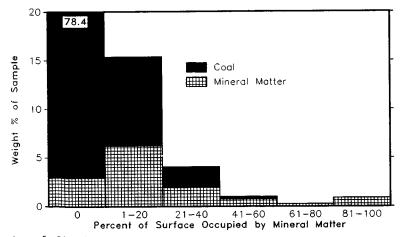


Figure 5. Distribution of coal and mineral matter in raw Upper Freeport coal as a function of particle surface occupied by minerals.

significantly reduce mineral content. Results of cleaning tests reported elsewhere (5) verified these predictions. Float-sink separations at 1.6 sp.gr. reduced the ash content by 57% with a 90% recovery, while froth flotation for 3 minutes resulted in only a 16% reduction in ash content with about the same recovery. Although these AIA results are quite preliminary, they show a strong general correlation with actual cleaning behavior.

### SUMMARY AND CONCLUSIONS

AIA-SEM provides insights into coal character and processing potential that are unavailable by other means. Many of the advantages of the technique stem from its ability to characterize coal and mineral particles, in-situ, on a microscopic level. Distributions of mineral matter as a function of particle size and mineral type are readily available and provide some indication of coal cleanability. Results are also now available showing the distribution of phases based on the weight fraction of mineral matter in the particle or based on the relative amount of surface of the particle occupied by mineral matter. These distributions can be related to processing behavior and can be used to explain, and possibly even predict, the recovery and quality of product under various cleaning conditions. The results are especially useful for detecting differences between various coals and for finding the reasons for unusual processing behaviors.

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